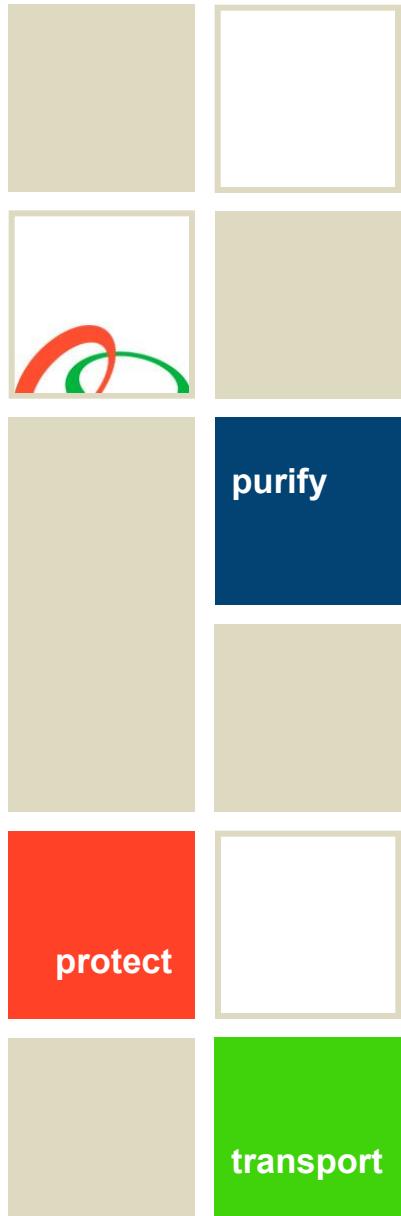


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## **SiC Design Guide** **Manufacture of Silicon Carbide Products**

**Mirror Technology Days**  
**June 7 to 9, 2010**  
**Boulder, Colorado**

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# Introduction to POCO Silicon Carbide

- History
- Objective
- Current Guide
  - Introductions to Ceramics
  - Chemical Vapor Conversion (CVC)
  - Materials selections
  - Design Basics Features
  - Manufacturing (from billet to precision machined assembly)
- The New Edition
  - Conversion bonding
  - Reaction-Bonding Silicon Carbide (RBSC) for large assemblies
  - Complex assemblies
  - Polishing

# History

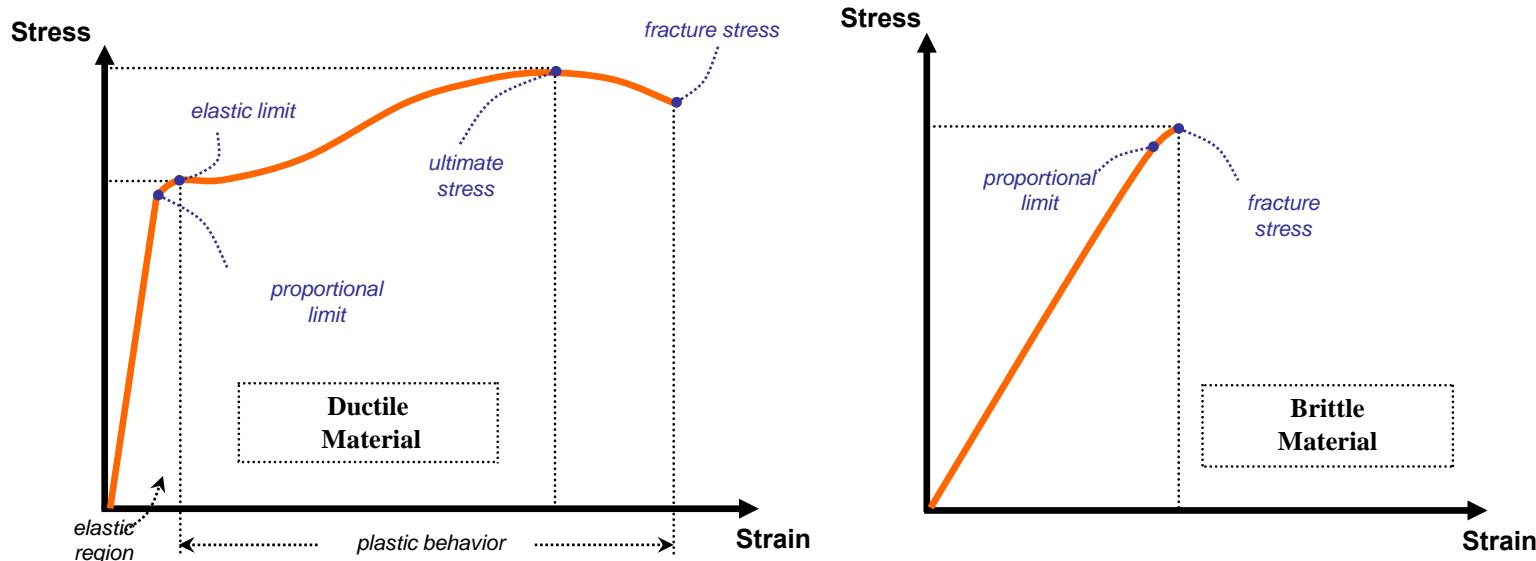
- Deliverable under an AFRL Phase I in 2006
- Revision was a SOW task under Phase II in 2008
- Contributing contracts:
  - another AFRL Phase II (closed)
  - an non-AFRL Phase II (closed)
  - an in-progress Army SBIR Phase II
  - an in-progress BAA
  - Other non-SBIR contracts

# Design Guide Objective

- These guidelines intended to pass on fundamental design principles and “best practices” that may assist engineers in component and assembly design.
- Through this guide, we hope to widen perceptions and reinforce the viability of silicon carbide as a versatile and cost effective material for many applications
- Useful to machinists, Sales persons, Manufacturing Support and other non-technical personnel
- Provide cost savings and reduced schedule throughout design transition to SiC
- Although an exhaustive effort was made to make this guide comprehensive, it is by no means all-inclusive

# Intro to Brittle Materials

- Limited ductility and complete lack of plastic behavior make ceramics inherently less “forgiving” than metals in some applications.
- Saying “strong but brittle” rings true and is a good adage to keep in mind, but does not detract from the material’s potential.

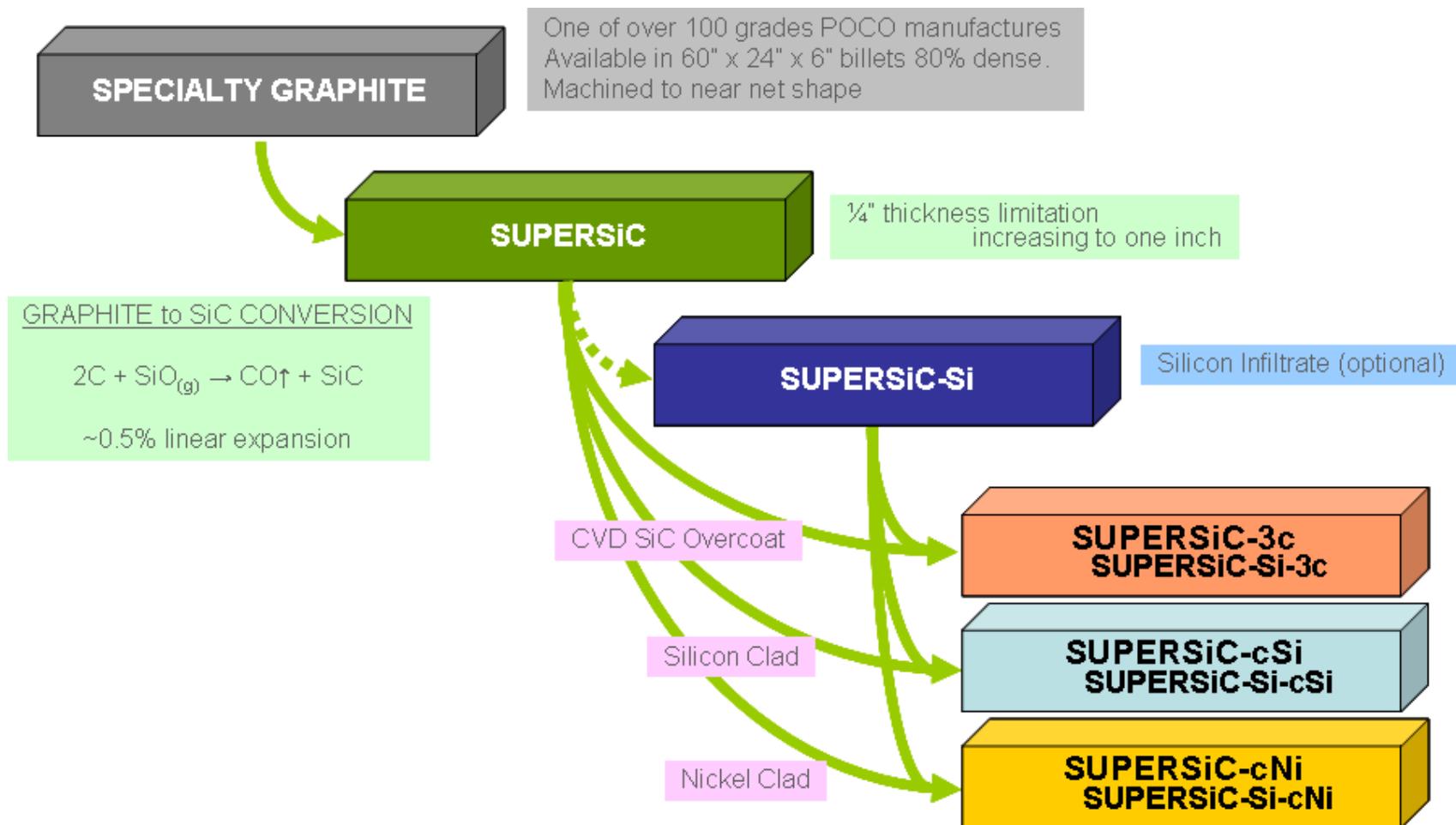


- SUPERSiC silicon carbide is an excellent choice for extreme-environment applications requiring superb thermal properties or the ability to achieve a high-quality optical finish on a material that satisfies structural needs.

# Intro to Ceramics

- Ceramics are a unique material family with performance unmatched by other materials.
- They are, however, brittle materials, and as such, engineers must gain an understanding of their nature to take full advantage of SUPERSiC when approaching design projects.
- These are the more significant characteristics:
  - Very high strength to weight ratio
  - Low ratio of tensile to compressive strength (these two properties are more comparable in metals)
  - Low and non-linear Coefficient of Thermal Expansion (CTE) characteristics
  - Virtually no yielding behavior prior to fracture mode failure
  - Relatively low impact toughness

# SUPERSiC Selections



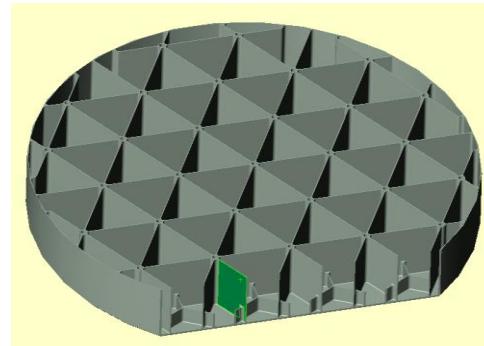
# Design Process Considerations

- Designing components from silicon carbide requires an understanding of application intent and expected performance.
- An engineer using SiC would want to develop parameters considering such issues as:
  - Is this application driven by strength or by stiffness target?
  - What is the application temperature range?
  - Will the part carry a static or cyclical load?
  - Is lightweight (low aerial density) critical?
  - What are the surface specifications?
  - Will the part be used for thermal conduction or insulation?
  - Are there corrosion concerns?
  - What gases may the part be exposed?
  - What pressures will the part be expected to operate?
  - Is there a preload on the part?
  - Are there electrical conductivity (grounding) requirements?
  - How will the part mate to its corresponding assembly?
  - What are the potential failure modes?
  - ... and many others

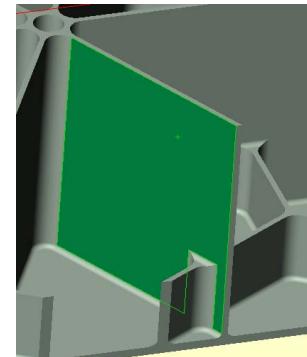
# Design Basics

Though there may be infinite design feature possibilities, following the intent of the few principle practices listed below will help save time and effort by reducing design iterations:

- Make attachment points robust, with load paths as straight as possible
- Design for compressive loads instead of tensile loads where possible
- Sharp edges may be prone to edge-chipping
- Typical aspect ratio of lightweighted structures is 26:1
- Component size is currently limited by our conversion furnace equipment size and/or our RBSC bonding furnace equipment
- POCO's graphite machining or SiC conversion processes do not produce burrs
- For some applications (highly erosive environments or where a part needs to be sealed) it may be desirable to apply a 100% dense, CVD SiC coating to encapsulate the SUPERSiC silicon carbide substrate

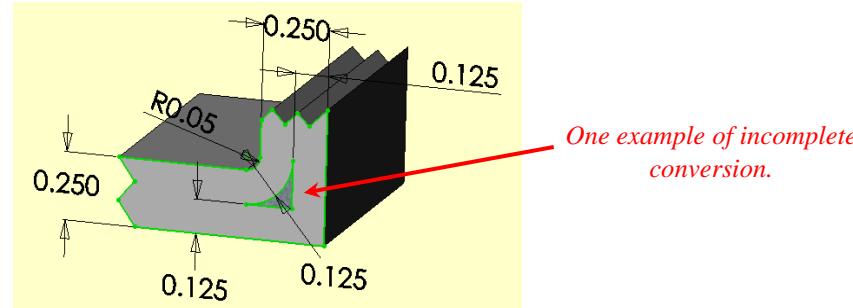


**Rib structure with aspect ratio 26:1**

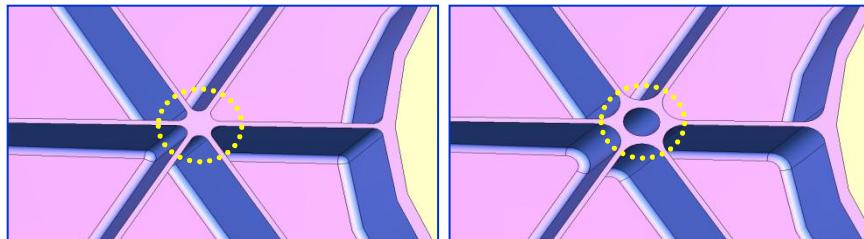


# Design for SiC Conversion

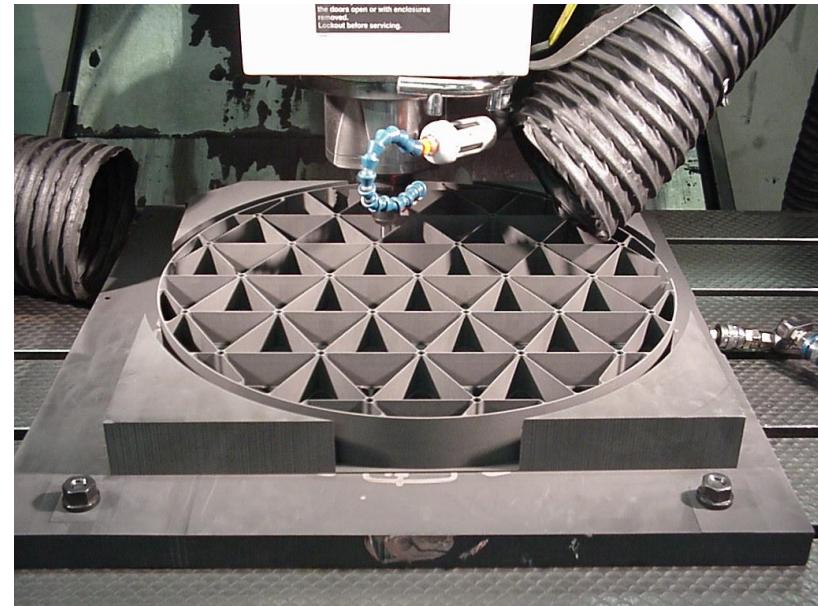
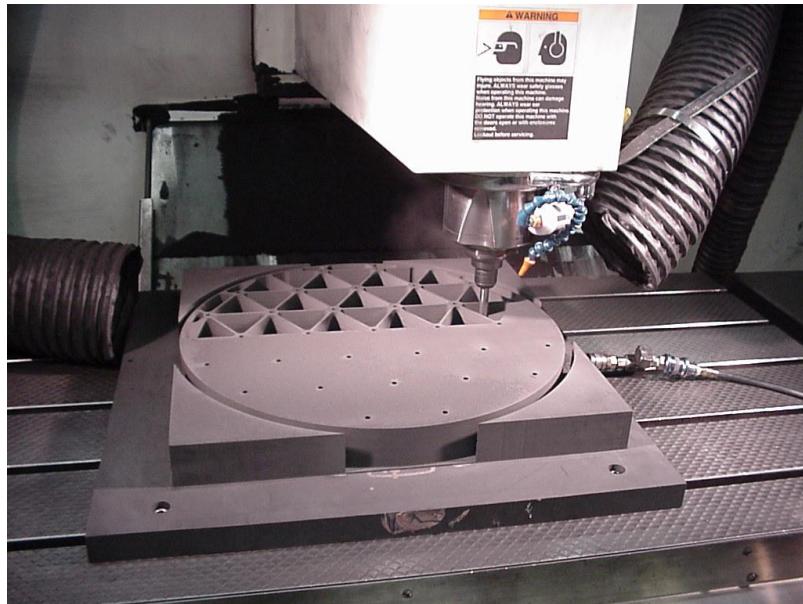
- Machined graphite components are converted in at high-temperature
- all parts are exposed to a gas mixture which promotes conversion
- **Successful conversion is dependant upon gas reaching and infiltrating all surfaces of the parts**
- **Infiltrating the porosity of all surfaces is dependant upon the geometric features designed into each part**
- Ensure maximum thickness at any point in the part is no greater than  $\frac{1}{4}$ "



- Preferred Radii, pockets, ribs

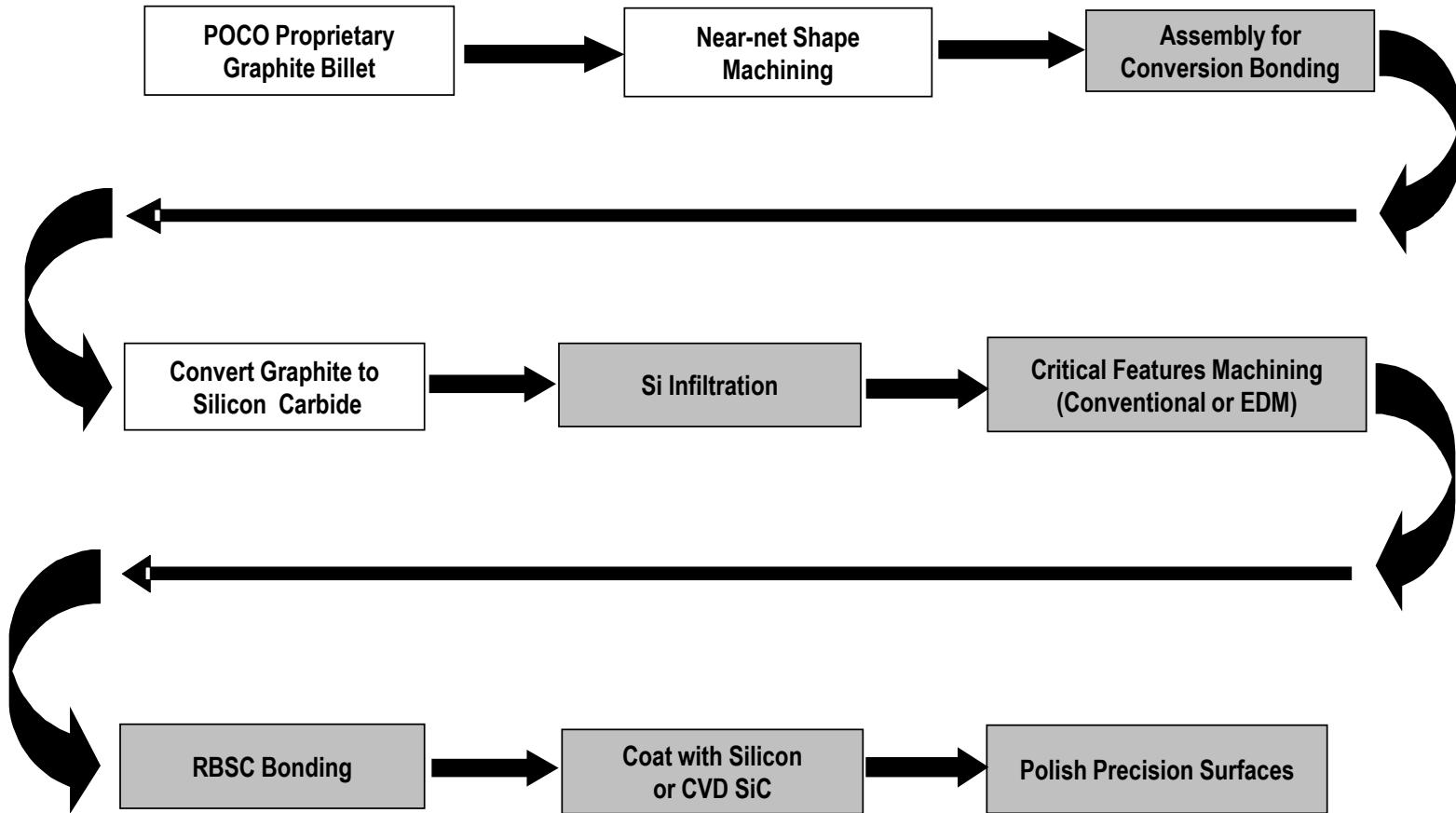


# Graphite Stage Near-Net Shape Machining



*These two photos show the progression of a mirror substrate rib-structure machined from a block of POCO graphite.*

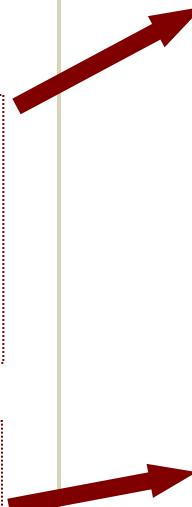
# SUPERSiC Silicon Carbide Manufacturing Process



# New for this Guide

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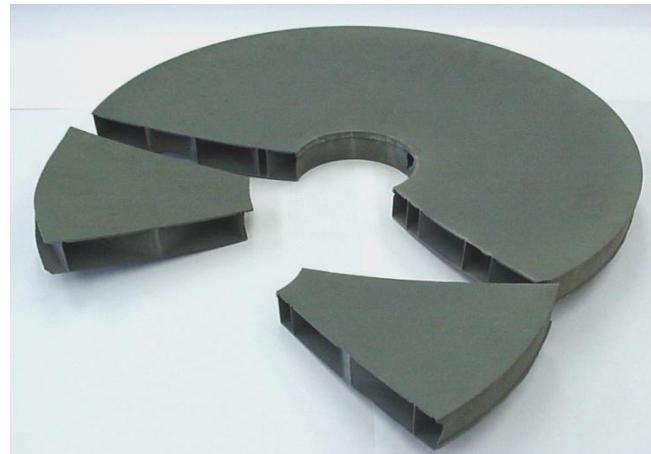
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# Creating Complex Structures

- Permanent Attachments
  - Conversion bonding basics
  - RBSC bonding basics (large structures)
  - Design for bonding
- Assembly joining methods
  - Helical Inserts
  - Direct contact threads
  - Solid Metal Inserts
    - Custom designed solid metal inserts
    - Adhesives

# Conversion Bonding

- The ability to assemble graphite pieces and subsequently convert the assembly into a monolithic SiC component **without** the use of adhesives is a critical feature of the POCO fabrication approach.
- It also adds significant design choices for the assembly of complex configurations.
- The resulting part is a complex, lightweight, rigid, closed-back substrate.
- **This adhesive-less permanent bonding process cannot be accomplished by any other silicon carbide manufacturing method.**
- The highly proprietary nature and complexity of this technology requires that POCO Engineering become an integral and early contributor to the design phase.
- The size limitation indicated earlier - any part that can fit into a cylindrical volume of 32 inches in diameter by 24 inches in height.

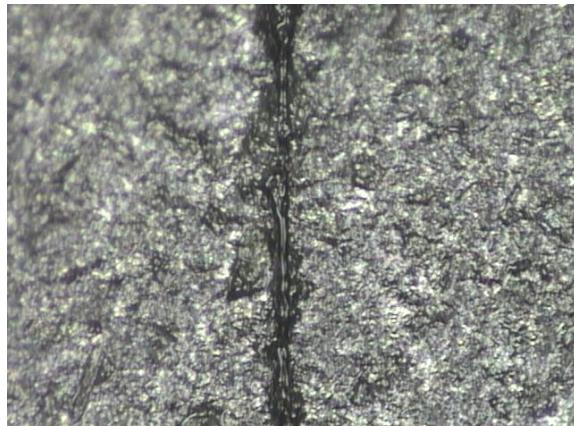


# Reaction-Bonding Silicon Carbide

- Developed for bonding large components and structures that are beyond the size limitations of POCO's conversion reactor
- Reaction-Bonding Silicon Carbide (RBSC) is a widely-used and proven means to permanently bond silicon carbide components and POCO has used this method for many years with great success
- Conversion bonding joins the graphite pieces **during** CVC and RBSC bonds components **after** they are converted to SiC
- A slurry is prepared and applied to the bonding surfaces and the components are joined and held together mechanically
- The assembly is placed in an oven and cured at a low temperature
- Once the slurry is hardened, the assembly is processed through high-temperature pyrolysis to convert the cured slurry to silicon carbide

# Design for Bonding

- Considerations include:
  - Surface preparation of the bonding interfaces
  - alignment of components
  - approaches to maintaining the assembly aligned during the high-temperature ( $>1500^{\circ}\text{ C}$ ) bonding processes
  - and the effects of post-bond machining.
- Adequate bondgap is When using RBSC bonding, proper bondgap must be considered (0.002" to 0.010")
- Bondgap design is critical for a robust interface design which may incorporate butt-joint contact or depth-controlled pockets for slurry injection

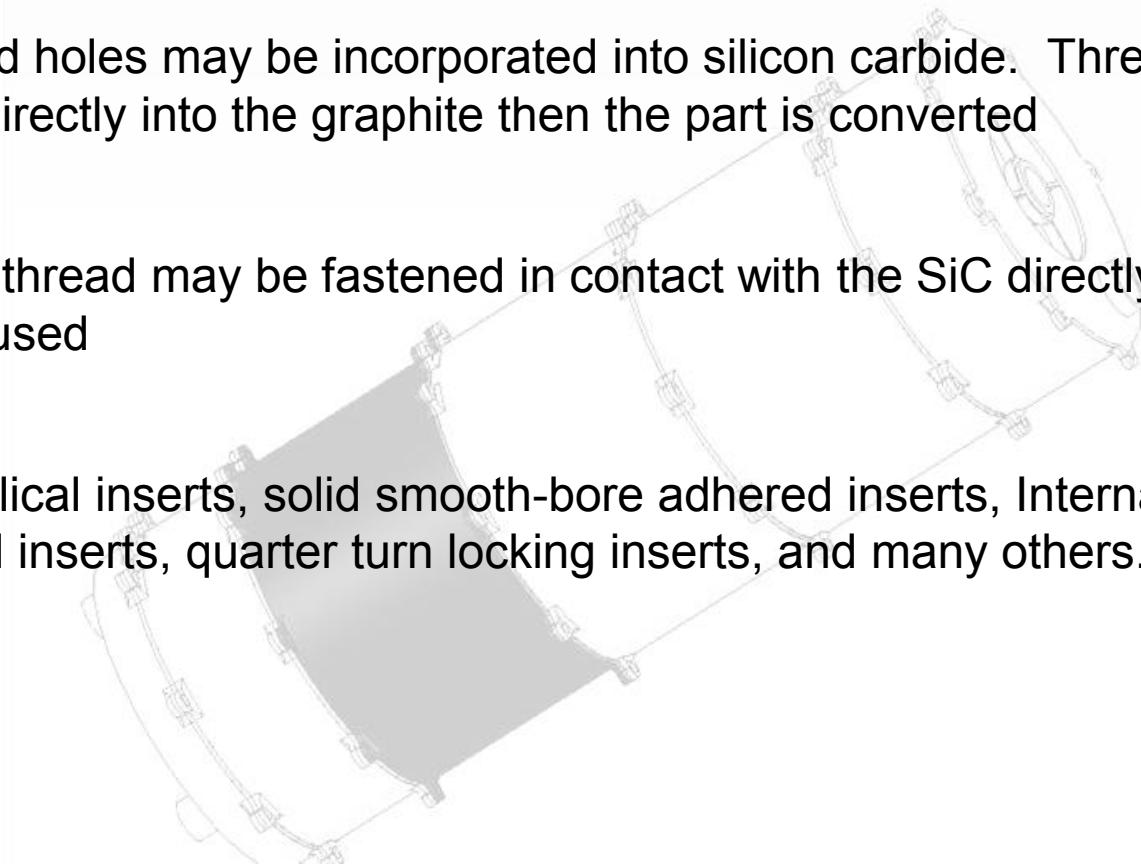


*In a magnified view, a 0.043mm MAX degraded edges can be seen on the seam, revealing also a complete fusion between surfaces in contact, due to the diffusion of silicon in SUPERSiC silicon carbide's porous body. A 0.076mm CVD SiC thick layer may be applied to bridge the seam and provide a uniform coating for polishing.*

# Assembly Joining Methods

POCO's SiC is able to utilize traditional mechanical joining methods

- Threaded holes may be incorporated into silicon carbide. Threads are tapped directly into the graphite then the part is converted
- The SiC thread may be fastened in contact with the SiC directly or an insert may be used
- Metal helical inserts, solid smooth-bore adhered inserts, Internal/external threaded inserts, quarter turn locking inserts, and many others.



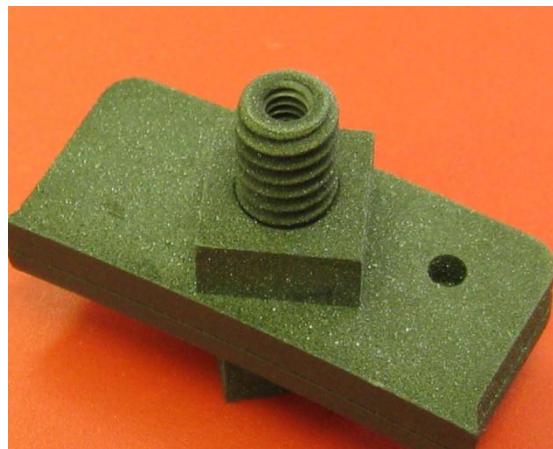
# Helical Inserts

- The most common insert technology for POCO SiC
- Used for many years in semiconductor components but is equally as reliable for other industries
- The metal helical is inserted directly into a tapped hole in the as-converted SiC part
- Testing shows that helical inserts provide strong mounting junction
- If the helical insert is installed deeper into the tapped hole (as much as double the depth) the resulting pullout strength can be increased by nearly 50%



# Direct Contact Threads

- Threads may be formed in the SiC component to a finished size that does not require an insert
- **The fastener in direct contact to the SiC threaded surface**
- Two ways to achieve the threaded surface: Conversion and EDM
- Conversion: Tap threads (not oversized) into the graphite then convert to SiC. Threaded holes are typically not designed for positional accuracy.
- If high positional precision is needed for an application, the threaded holes may be Electric Discharge Machined (EDM) after SiC conversion
- It is not uncommon for the EDM process to provide an accuracy down to 0.0005”.



*This component is unique in that it shows four pieces conversion bonded together. Two graphite flat plates were held together with a graphite nut and bolt then all the components were conversion bonded to produce one SiC part. After conversion, EDM was used to machine an internal 4-40 thread through the bolt center allowing for further assembly joining.*

# Solid Metal Inserts

- In some cases, provides a stronger mounting feature than helical inserts
- Fasteners tend to fail in shear under torque and tensile testing before substrate, insert, or adhesive fails
- Can provides a pad for precision machining (coplanarity) without SiC grinding

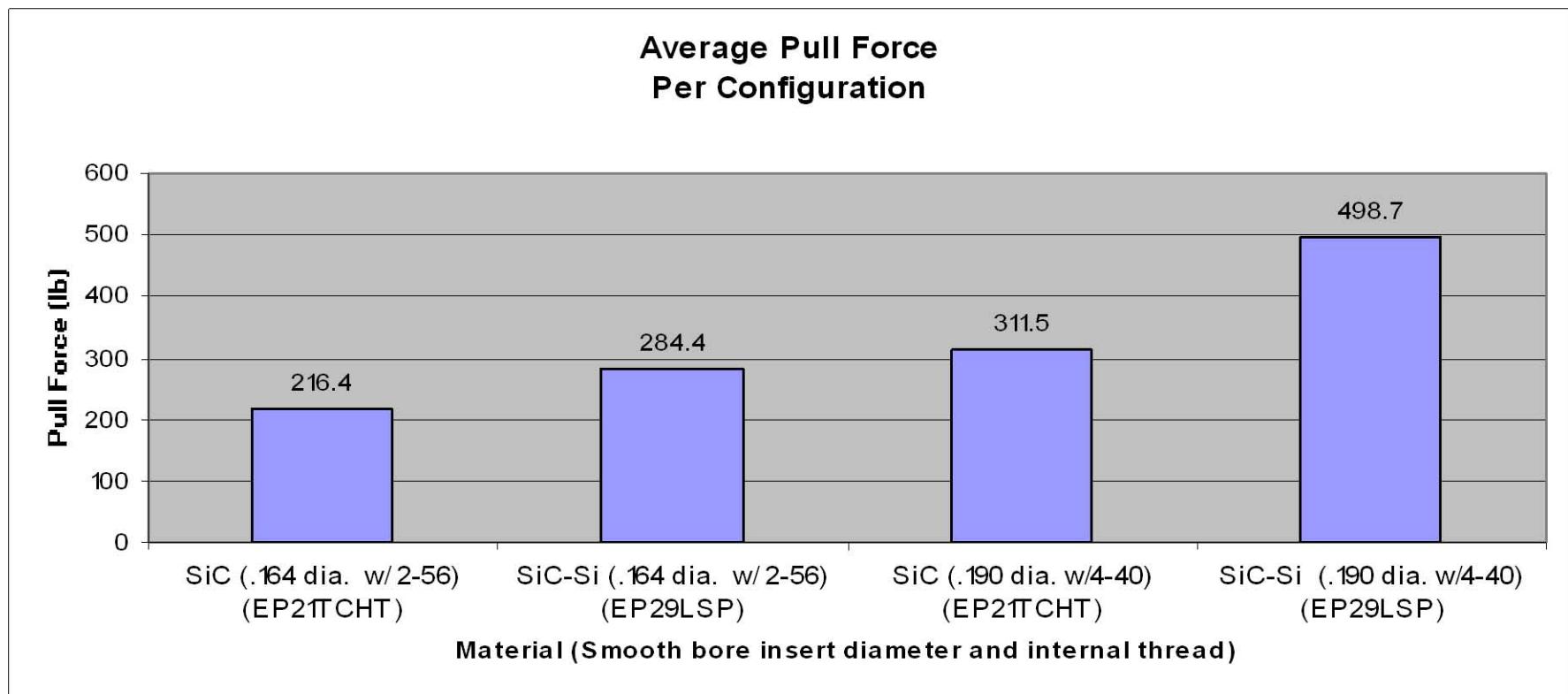


# Adhesives

- Cryogenic conditions would present challenges that would require an adhesive that compensates for the CTE-affected physical changes
- Invar® type alloys are recommended for cryo applications
- Adhesives may be used as the full bonding media for smooth-OD inserts or as a staking mechanism („thread lock“) only for threaded inserts
- Master Bond® EP21TCH and EP29LSP have service temperatures as low as 4 Kelvin and are resistant to cryogenic shock
- EP21TCHT works well with porous SUPERSiC and non-porous SUPERSiC-Si material
- EP29LSP is better suited for non-porous SUPERSiC-Si material due to its viscosity
- Hysol® 9394 is a good all-around adhesive though not favored for extreme cryo conditions
- Several other adhesives are also available

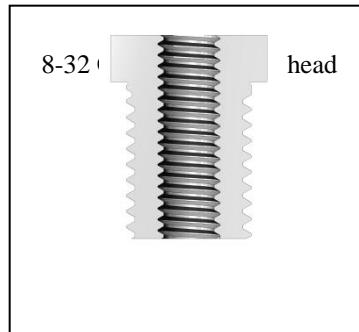
# Strength of Adhered Solid Metal Inserts

The following chart contains information obtained by testing smooth-OD Invar type inserts into SUPERSiC and SUPERSiC-Si.



# Thread Selection and Strength

- POCO has conducted many tests to characterize the strength of threaded holes with either bare silicon carbide engagement or using inserts of choice.
- Torque measurement are provided in the guide. However, it should be noted that POCO engineering concurs with most industry opinion that torque provides results that are a poor indication of a joints strength capabilities due to the many factors including indeterminate friction factors.
- Our intent in conducting this research is to continually collect value added properties information and pass it on to customers in addition to expanding the variety of design and manufacturing techniques in our application toolbox.



Typical Internal Threads (UNEF, UNF, UNC)		Typical External Threads	
2	.086	8-32	.164
4	.112	10-24	.190
6	.138	12-24	.216
8	.164	1/4-20	.250
10	.190	5/16-24	.3125
1/4	.250	3/8-24	.375

# Coatings for Optics

- Coatings for chemical protection, increased oxidation and electrical resistance, and „polish-ability“
- Three coating technologies: Silicon, Nickel, and CVD SiC
  - Silicon Cladding
    - for optical face (pads optional)
    - lower cost than CVD SiC for non-flat surfaces
    - masking fixtures required for complex components, selective surface cladding
    - easier material to machine by conventional or SPDT (tool wear beyond 8" diameter is a concern)
    - Conventional buffing may follow SPDT to produce final finish
    - Cladding of mounting pads is an option to lapping of SiC to achieve coplanarity
  - Nickel
    - Nickel can be plated on the conductive SUPERSiC silicon carbide substrate, but for room temperature applications only

# Coatings for Optics-cont'd

## – CVD SiC

- Applications requiring sealed or impervious surfaces
- Strength values differ little from the SiC substrate
- Selected for flat optics
- High tool wear is common and is a major contributor to increased process cost.
- Accurate positioning of critical features such as attachment faces and tabs may be accomplished by grinding.
- Tolerances resulting from precision grinding are typically  $\pm 0.001$  inches, but can be better than  $\pm 0.0002$  inches depending on part geometry.
- Polished to very fine finishes and accurate surface contours
- Considerably more expensive than machining graphite
- The deposited coating offers the chemical resistance of silicon carbide against many harsh chemical environments and the low surface area of the coating reduces the bulk oxidation rate in high temperature environments.
- The high purity coating can also act as an electrical insulator.
- Typical coating thicknesses are 0.003 and 0.008" with double coatings of each thickness available

# Coatings Comparison Chart

- In general, CVD SiC is selected for flat optics and silicon or nickel for non-flat optical surfaces.
- Typical finish and figure values are listed in the table below:

SUBSTRATE	4" DIAMETER FLAT		8" DIAMETER ASPHERE		
	MATERIAL	FIGURE <sup>[1]</sup>	FINISH	FIGURE	FINISH
CVD SiC		$\lambda/12$ p-v	<5 Å rms	$\lambda/8$ p-v	<10 Å rms
Silicon		$\lambda/8$ p-v	<10 Å rms	$\lambda/4$ p-v	<20 Å rms
Nickel		$\lambda/2$ p-v	<40 Å rms	not available	not available

[1]  $\lambda = 6328 \text{ \AA}$

# Optical Quality Polishing

- Substrate preparation
  - Preparation for achieving an optical finish begins with selection of the appropriate converted substrate made from POCO's porous silicon carbide, SUPERSiC or silicon infiltrated SUPERSiC-Si (SUPERSiC-3C or SUPERSiC-Si-3C, respectively, when coated), depending on application preferences
  - Prior to CVD coating, fine grinding, or contour grinding, of the substrate material is performed in order to achieve a profile-of-surface of within 0.001~0.002 inches on the optical face
- Figure and finish
  - After contour grinding comes the final, optical polishing step
  - POCO does not perform this polishing operation
  - SiC has been polished by multiple methods to highly accurate surface figure and finish.
- In addition to flat mirror, we have worked with our partners to developed processes for polishing aspherical, concave, and convex shapes
- POCO continues work with multiple optical finishing suppliers and good results have been achieved by both conventional and unconventional methods

# SiC Design Guide

## Manufacture of Silicon Carbide Products

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